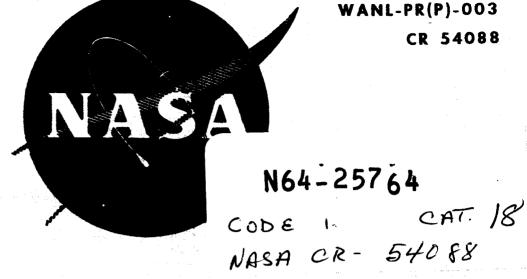
CR 54088



DETERMINATION OF THE WELDABILITY AND ELEVATED TEMPERATURE STABILITY OF REFRACTORY METAL ALLOYS

Third Quarterly Report

G. G. Lessmann and D. R. Stoner

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION **LEWIS RESEARCH CENTER UNDER CONTRACT NAS3-2540**

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nou onuclear Laboratory **Westinghouse Electric Corporation**

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by

G. G. Lessmann

and

D. R. Stoner

THIRD QUARTERLY REPORT

Covering the Period

December 21, 1963 to March 21, 1964

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION Contract NAS 3-2540

Technical Management
Paul E. Moorhead
NASA - Lewis Research Center

Astronuclear Laboratory
Westinghouse Electric Corporation
Pittsburgh 36, Pa.



FOREWORD

This report describes work accomplished under Contract NAS 3-2540 during the period December 21, 1963 to March 21, 1964. This program is being administered by R. T. Begley of the Astronuclear Laboratory, Westinghouse Electric Corporation. G. G. Lessmann and D. R. Stoner performed the experimental investigations.

P. E. Moorhead of the National Aeronautics and Space Administration, is Technical Manager of this program.



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I. INTRODUCTION

This is the Third Quarterly Progress Report describing work accomplished under Contract NAS 3-2540. The objective of this program is to determine the weldability and long time elevated temperature stability of promising refractory metal alloys in order to determine those most suitable for use in advanced alkali-metal space electric power systems. A detailed discussion of the program and program objectives was presented in the First Quarterly Report.

A chronological outline of this study is shown in Figure 1, while those alloys to be included in the investigation are listed in Table 1. Each alloy will receive essentially the same evaluation, though as the program evolves certain alloys may require tests tailored to unique responses to test conditions. Alloys will be tested in the form of 0.035 inch sheet and 0.375 inch plate, with the exception of W-25% Re, unalloyed W, and Sylvania "A" which will be evaluated only in sheet form. The inert gas shielded arc and electron beam welding processes will be used.

Process and test controls employed throughout this program emphasize the important influence of the interstitial elements, carbon, oxygen, and nitrogen, on the properties of refractory metals and their alloys. Stringent test procedures are required, including continuous monitoring of the welding chamber atmosphere for oxygen and water vapor levels, electron beam welding at low pressures, aging in furnaces employing hydrocarbon free pumping systems providing pressures less than 10-8 Torr, and chemical sampling following successive stages of the evaluation for verification of these process controls.



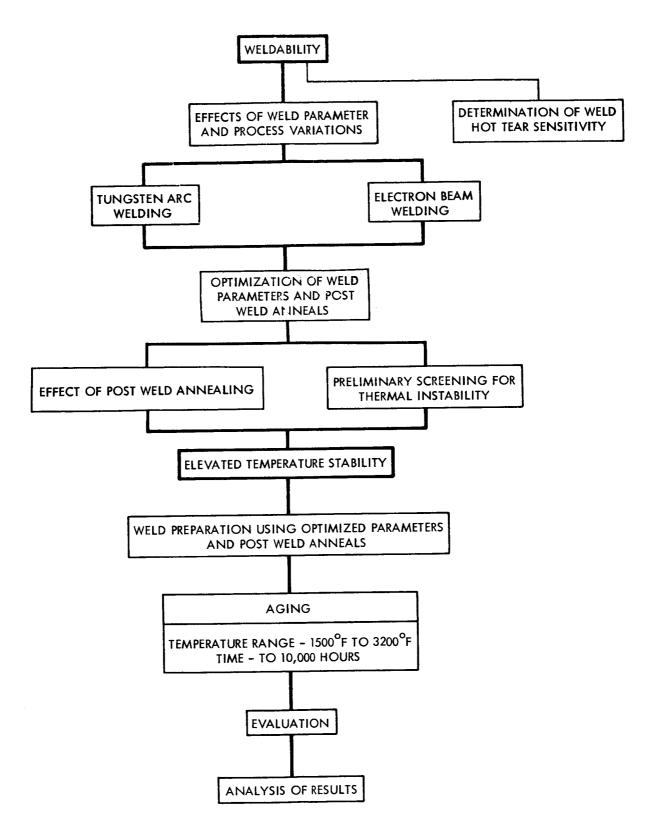


FIGURE 1 - Chronological Program Outline



TABLE 1 - Alloys Included in the Weldability and Thermal Stability Evaluations

Alloy	Nominal Composition Weight Percent
AS-55	Cb-5W-1Zr-0.2Y-0.06C
B-66 1/	Cb-5Mo-5V-1Zr
C-129Y	Cb-lOW-lOHf+Y
Cb-752	Cb-10W-2.5Zr
D-43 V	Cb-10W-1Zr-0.1C
FS-85	Cb-27Ta-10W-1Z r
SCb-291 ¥	Cb-lOW-lOTa
T-111	Ta-8W-2Hf
T-222	Ta-9.6W-2.4Hf-0.01C
Ta-10W	Ta-lOW
W-25 Re	W-25Re
W	Unalloyed
Sylvania "A"	W-0.5Hf-0.02C

Note: All alloys to be from arc-cast and/or electron beam melted material except Sylvania "A"



II. SUMMARY

Purchase orders have been placed for the thirteen alloys listed in Table 1 and have been filled for eight alloys. The delivery status of alloys is shown in Table 2. Test data for seven of these are included in this report. Incoming inspection of this material included visual, dimensional, chemical, and metallographic inspection.

A study of the effects of weld parameter variations was initiated on seven alloys. The electron beam welding effort of this phase was completed for FS-85 and Ta-10W. TIG welding was initiated for these and for B-66, C-129Y, Cb-752, D-43, and SCb-291. Bend test results on welds and base metal are summarized in Figure 17. Analyses and discussion of the welding data will be deferred for a later report when the weld test series has been completed.

Restraint weld tests were completed for the available alloys. Bead-on-plate patch tests for sheet material and circular groove restraint tests for plate material were used. These tests were described in earlier reports.1,2

A double "U" butt weld joint configuration was test welded for each alloy and found to be satisfactory for welding the 3/8 inch plate.

Further modification of the TIG weld atmosphere monitoring equipment was required. This system is now working satisfactorily.

TIG weld chamber tooling was installed and performed satisfactorily during initial weld tests. Copper clamp bar inserts were troublesome at narrow clamp spacing and are being replaced with molybdenum inserts.

An evaluation was made of the effect of variation of vacuum pressure in the range of 10^{-3} to 10^{-5} Torr on EB weld contamination. These results are being compared with the best TIG welding atmosphere. No differences were noted in pickup of contaminants in the EB welds. Bend test results did not reflect any difference. This type of process check will receive continued emphasis throughout the program.

A series of weld chamber glove tests was initiated in an effort to identify the most suitable glove material. The monitoring system proved most useful in this respect since it permits measuring the decay of atmosphere quality as a function of time for different glove types. Neoprene, butyl rubber, and polyvinyl chloride gloves have been tested. All of these have limitations. The polyvinyl chloride gloves were unsatisfactory. Additional glove tests are anticipated.



Four ultra-high vacuum aging furnaces for use in the thermal stability studies were checked out at the factory, delivered, and installed. These units are described in this report.



TABLE 2 - Alloy Procurement and Delivery Schedule

Placed
1/29/64 5/1/64
8/19 8/29 10/18
9/20 10/2 11/30
9/19 10/21 11/30
8/17 9/3 11/8
8/12 8/22 1/30/64
9/17 10/2 11/30
8/16 9/27 10/28
9/25 10/21 1/15/64
8/12 8/22 9/30
11/26 2/10/64 4/1/64
2/19/64 4/16/64 6/15/64
5/15/64

1. Sheet Material produced by Fansteel under Contract NOw-63-0231-c and furnished to this program as transferred government owned material.



III. TECHNICAL PROGRAM

A. ALLOY PROCUREMENT

The procurement status of the alloys included in this program is shown in Table 2. All alloys ordered are being produced from arc cast (or EB melted) ingots except Sylvania "A" which is consolidated by powder metallurgy. The tungsten base alloys are being evaluated in sheet form only. The investigation of unalloyed tungsten and Sylvania "A" will be more limited in scope than for the other alloys because of the special handling problems associated with the brittleness of these alloys. All material received has been satisfactory as determined by visual, dimensional, metallographic, and chemical (interstitials only) inspection except T-111 wire which was found to be contaminated. Chemistry hardness and grain size data for as-received material are summarized in Tables 3 and 4. Photomicrographs of the as-received material are shown in Figures 2 through 6. Alloys not shown were included in the second quarterly report. All plate and sheet material, except D-43 plate, is in the recrystallized condition.

B. WELDING EVALUATIONS

1. Procedures

a. <u>TIG Welding</u>: Tungsten inert gas shielded arc welding is being conducted in a vacuum purged and backfilled weld chamber. All sheet butt welds are being made automatically by fusion only without the addition of filler wire. Plate welds will generally be made manually although semiautomatic techniques will also be explored. In both cases high frequency is employed for arc initiation.

Process variables have been minimized within practical limits consistent with the objectives of obtaining a comparative weldability rating for each alloy and a measure of alloy sensitivity to weld process variation. Emphasis in the welding evaluation is directed towards identifying effects arising from variation of weld freezing rate, cooling rate, and unit weld length heat input rather than current, speed, and voltage per se. For this purpose the number of extraneous weld variables has been minimized. Electrode configuration, arc gap, and shielding gas (helium) are being kept constant. This permits variation of weld speed (freezing rate), clamp spacing (cooling rate), and weld size (arc current). Straight polarity DC current is being used. The same power supply will be used throughout this evaluation.

The vacuum purged weld chamber and power supply have been described in an earlier report. In preparation for welding, the chamber is evacuated by overnight pumping to $5-9 \times 10^{-6}$ Torr and checked for a leak rate of less than 4×10^{-5} Torr per minute. Backfilling is accomplished rapidly (within



33 538 83**8** 8 8 8 Check Chemistry 28 130 130 130 200 34 34 K4555 865 १८८ १८८ १८८ १८८ १८८ १८८ १८८ १८८ 88 INBLE 3 - Chemistry of As-Received Material 28.1 27.61 28.0 9.83 9.6 9.2 Bal. Bal. Bal. Bal. Certified Analysis (Avg.) 25.6 4.89 4.89 5.61 4.85 5.17 10.25 10.10 9.5 10.25 1.7 2.01 1.00 1.00 0.92 0.93 0.94 0.95 0.95 2.90 2.90 52165 52208 52183 Plate Sheet Wire Plate Sheet Wire Sheet Wire Sheet Wire Plate Sheet Wire Plate Plate Plate Sheet Wire Mire Mire Sheet Mire SCb-291 Cb-752 C-129Y T-111 D-43 W-Re

8



TABLE 4 - Hardness and Grain Size of As-Received Material

Alloy	Form	Hardness DPH	ASTM Grain Size
B-66	Plate Sheet	225 219	6 10
D-43	Plate Sheet	202 220	5
FS-85	Plate Sheet	205 190	7 8
Cb-752	Plate Sheet	204 205	8 8 - 9
SCb-291	Plate Sheet	160 175	6
C-129Y	Plate Sheet	218 185	10
T-111	Plate Sheet	223	6–7
Ta-10W	Plate Sheet	197 221	8 6–7



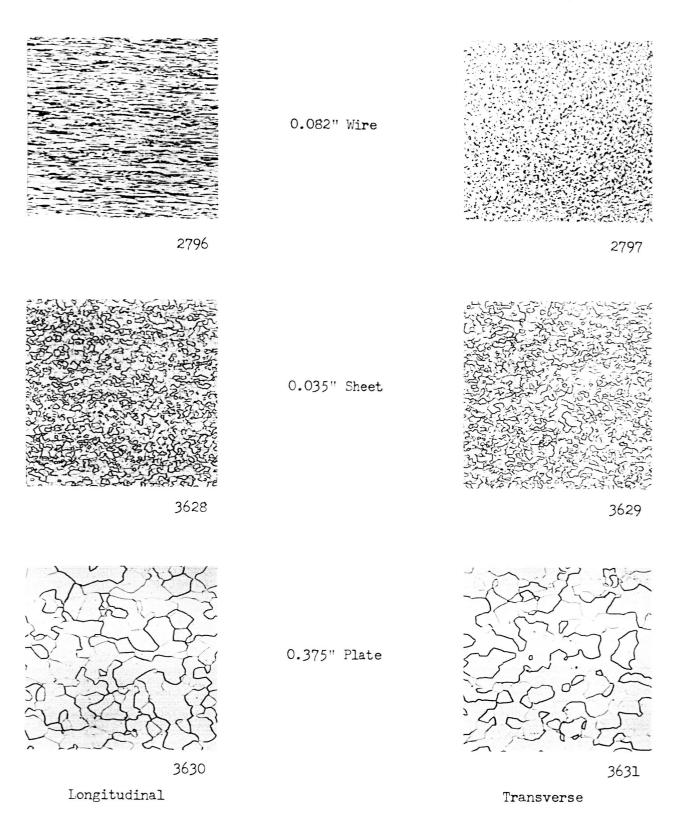


FIGURE 2 - Microstructures of As-Received B-66, 100X (HNO₃-NH₄F·HF Etch)



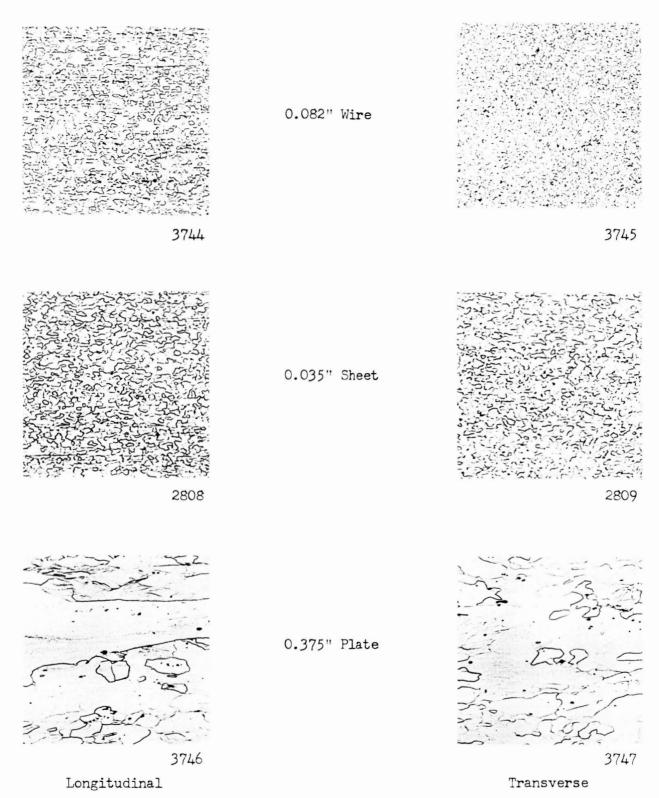


FIGURE 3 - Microstructures of As-Received C-129Y, 100X (HNO₃-NH₄F·HF Etch)



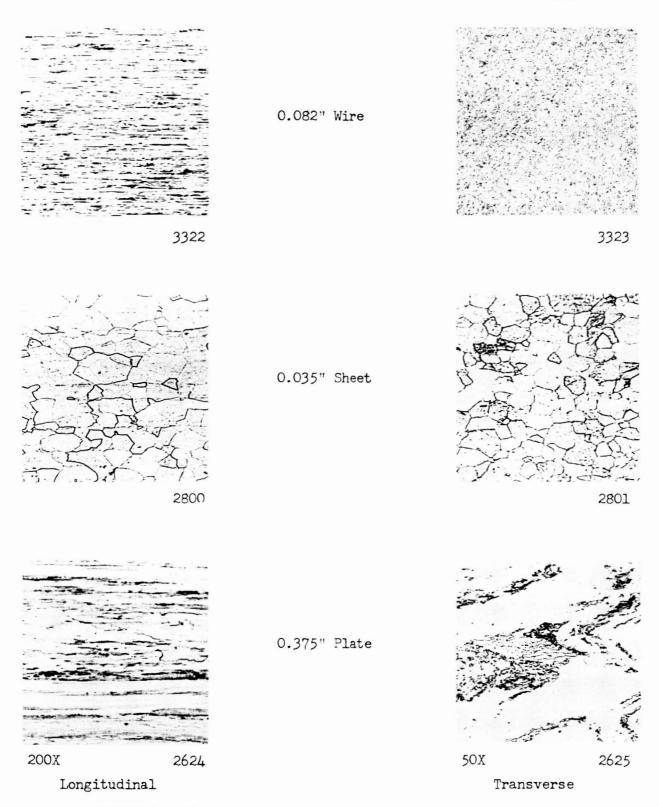


FIGURE 4 - Microstructures of As-Received D-43. 100X Except As Noted (HNO3-NH4F.HF Etch)



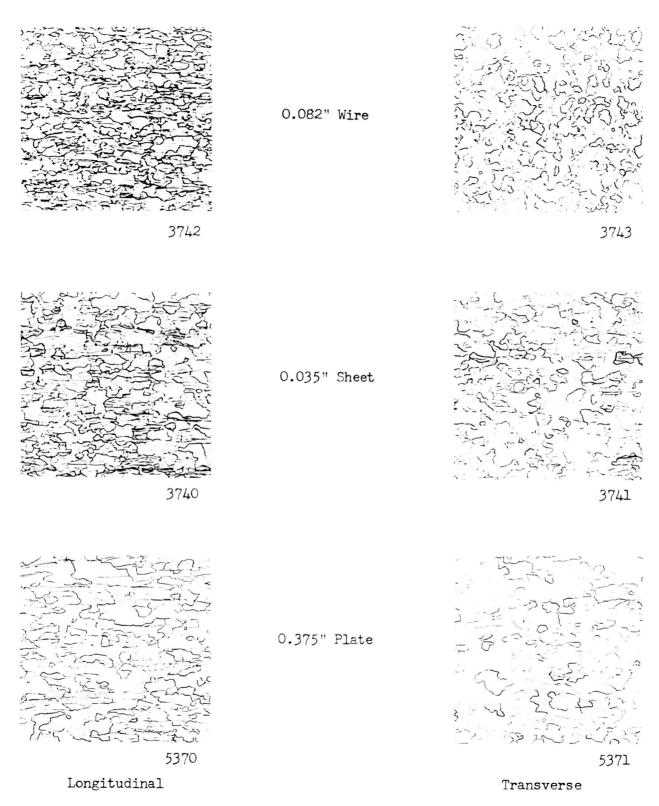


FIGURE 5 - Microstructures of As-Received FS-85, 100X (HNO₃-NH₄F·HF Etch)



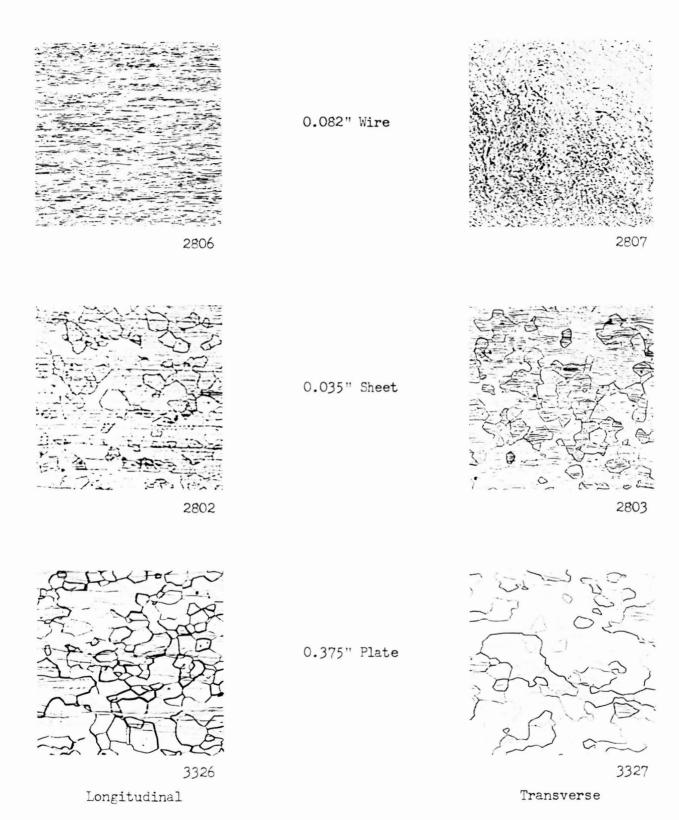


FIGURE 6 - Microstructures of As-Received SCb-291, 100X (HNO3-NH4F.HF Etch)



one minute) to minimize contamination. The weld chamber atmosphere is monitored during welding for oxygen and moisture content. The oxygen level of the weld atmosphere immediately after backfilling is consistently less than 5 ppm (generally 1 to 3.5 ppm) and does not increase significantly during a normal weld run of two to three hours. Backfilled moisture levels are generally between 0 and 0.5 ppm and rise continuously to a level of 3-5 ppm during normal weld runs. The monitoring system is described in detail in previous reports, 1,2 while the most recent performance data are presented in Section III, C of this report.

The sheet butt weld set-up is shown in Figure 7. The welding electrodes are machine ground to the dimensions shown. The electrode extension from the torch shank as well as the arc gap is fixed. The actual weld fixturing, water cooled torch, and drive table are shown in Figure 8. The drive table is fabricated from aluminum, the clamp down fixture from stainless steel with copper or molybdenum inserts, and the torch from copper. Torch cooling water is supplied through flexible convoluted stainless steel tubing. The tubing is silver soldered to the torch and feed throughs. The manual welding torch (not shown) consists of a water cooled copper electrode holder to which water is also furnished by flexible stainless steel hose. Welding current is conducted in concentric layers of shielding cable attached to the outside of the flexible tubing. "Tygon" is used for electrical insulation.

Sheet specimens of columbium and tantalum alloys are prepared for welding by shearing and pickling in an acid solution containing 25% concentrated nitric acid, 25% hydrofluoric acid (50% concentration) and 50% tap water (by volume). Approximately 0.5 to 1 mil per surface is removed. Immediately after pickling, all material is rinsed for one minute in cold circulating tap water, then in hot water, then in distilled alcohol and hot air dried. Following cleaning and pickling, only clean cotton gloves are used for handling these alloys. Cleaned and pickled specimens are stored in new polyethylene bags or wrapped in new aluminum foil.

Plate specimens are prepared for welding by cutting and machining and then pickling and handling in the same manner as sheet material. The selected joint preparation is shown in Figure 9. This configuration has proven satisfactory for all alloys received to date. The root pass is made at zero clearance without the addition of filler metal. Additional passes, two for columbium base alloy and three for Ta-10W, with filler wire added manually on each side complete the butt weld. The filler wire diameter is 0.082 inches. Specimen flatness is controlled by alternate welding on opposite sides of the weld joint. A typical weld schedule for the plate butt welds is shown in Figure 10. Plate butt weld test samples for seven alloys are shown in Figures 37 through 40 along with circular groove restraint test specimens. No difficulty was encountered in welding these except the root pass in B-66 required heavy end tacks to avoid hot tearing. Excellent joint flatness was



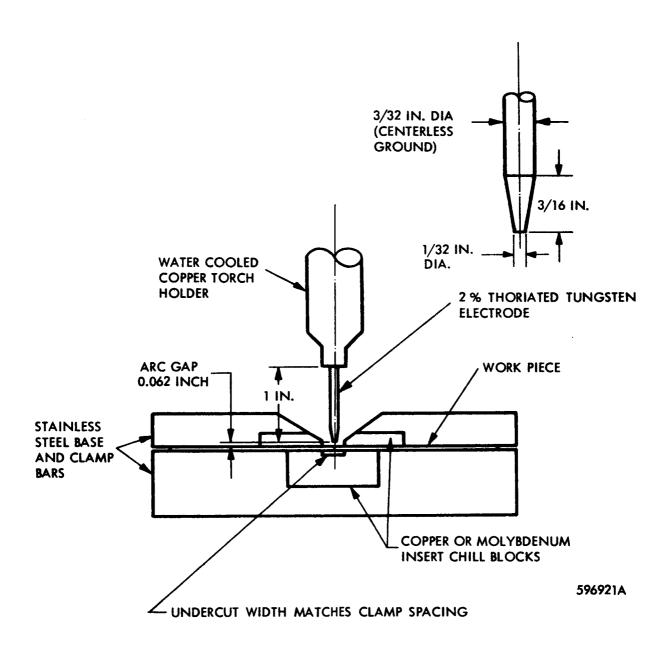


FIGURE 7 - Weld Set-up For TIG Sheet Butt Welds



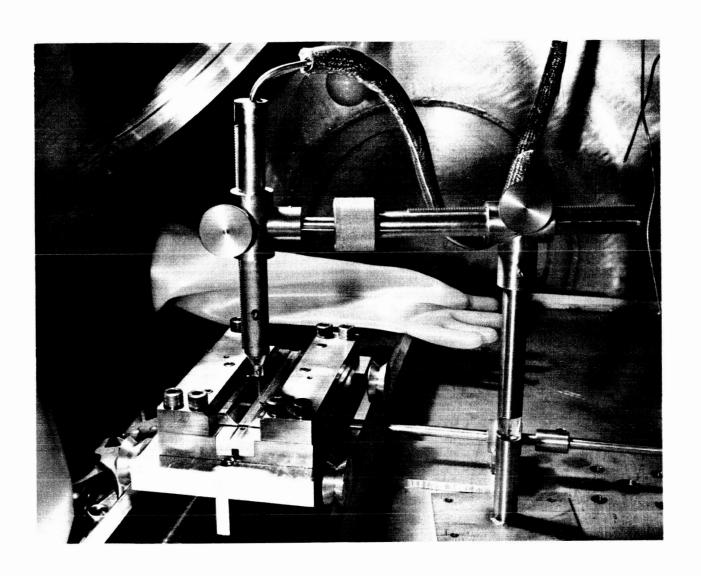
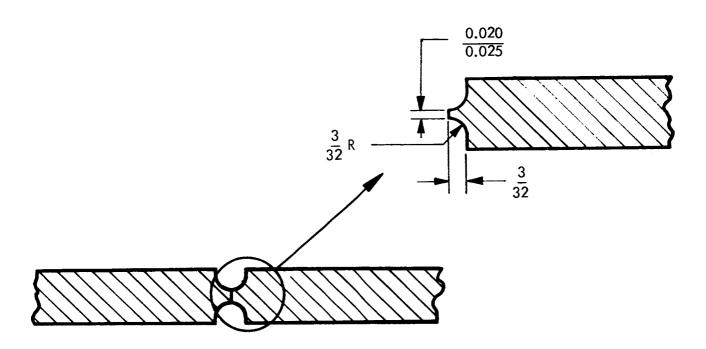


FIGURE 8 - TIG Butt Weld Fixtures





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FIGURE 9 - Plate Butt Weld Joint Configuration



WELD 321 - SCb-291 Butt Weld, 3/8 Inch Plate. Wire Required 11 Inches x 0.082 Inch Diameter

- Tack welded in center and at one end of joint. Positioned in clamp down fixture. 155 amperes.
- 2. Fusion pass on side #1. 300 amperes. Continuous weld from one end. One side of joint clamped, other side cantilevered.
- 3. Fusion pass on side #2. 280 amperes. Specimen supported along each edge on copper blocks. Continuous weld.
- 4. First filler pass on side #2. 300 amperes. Continuous weld.
- 5. First filler pass on side #1. 300 amperes. Continuous weld.
- 6. Second filler pass on side #2. 280 amperes. Continuous weld.
- 7. Second filler pass on side #1. 280 amperes. Continuous weld.

Final Monitor Readings: 2.4 ppm H₂O, 3.4 ppm O₂

FIGURE 10 - Weld Schedule for SCb-291 Butt Weld in 3/8 Inch Plate. Typical for Columbium Base Alloys.



maintained in producing these joints. Dye penetrant tests were all negative.

b. Electron Beam Welding Procedures: A Hamilton-Zeiss 2KW high voltage electron beam welder is used for all electron beam welding. Since high voltage welders (150KV) operate with most effective beam size control in the higher voltage ranges, initial parameter evaluation included accelerating voltages of 100 and 150KV. Preliminary weld examination indicated that the accelerating voltage range had no significant control of the weld bead shape in 0.035 inch sheet, consequently a constant voltage was picked to allow a greater choice of other parameters.

Following preliminary welding trials, the plan of parameter evaluation shown in Figure 11 was adopted. This combination of welding parameters produced a general trend of increasing power input per unit length of weld with decreasing weld speed as shown in Figure 12. This trend is expected to produce a distinct change in the freezing rate of the weld metal, and coupled with a significant change in chill spacing is expected to produce a continuously varying size and thermal history of the heat affected zone. Aluminum clamps, machined with weld centerline to chill spacings of 0.250 inch and 0.095 inch, are shown in Figure 13. Six welding fixtures are available, permitting economical use of the operator and equipment.

All sheet welds were butt welds of two 1/2 inch wide by 6 inch long specimens. The narrow beam size, 0.010 inch, required machined edges to prevent "burn through" of slightly misaligned edges. Following machining, all specimens were cleaned, pickled, and handled using the same procedures as for TIG weld specimens.

Welding parameters were developed by material penetration trials aimed at measuring the amperage required for 100% penetration. Power settings for the actual weld passes were increased 10% to insure complete penetration under the normal variation in welding conditions.

At each power setting the beam was focused optically prior to welding to achieve the smallest beam size possible. Transverse and longitudinal beam deflection, Figure 14, was similarly measured on a tungsten target. Using this power and beam adjustment procedure, variations in weld bead size and penetration between different alloys and welding parameters will be minimized.

Normal full penetration electron beam welds will produce a vapor deposited coating of the clamp material on the underbead side of the welds. Backup strips of the same composition as the weld specimens are used to minimize this source of contamination.

Early welds were made at a chamber pressure prior to welding of 2 x 10^{-5} Torr, while the balance of the Ta-10W and FS-85 welds were made at 5 x 10^{-6}



Travel Speed (ipm)	Deflection (inches)
100	No deflection
100	0.050 longitudinal
50	0.050" longitudinal
25	0.050" longitudinal
15	0.050" longitudinal
15	0.050" transverse
0.250" & to Clamp Spacing	0.094" & to Clamp Spacing

Accelerating Voltage Constant at 150 KV Amperage Adjusted for 100% Weld Penetration

FIGURE 11 - General Outline of Electron Beam Weld Parameters



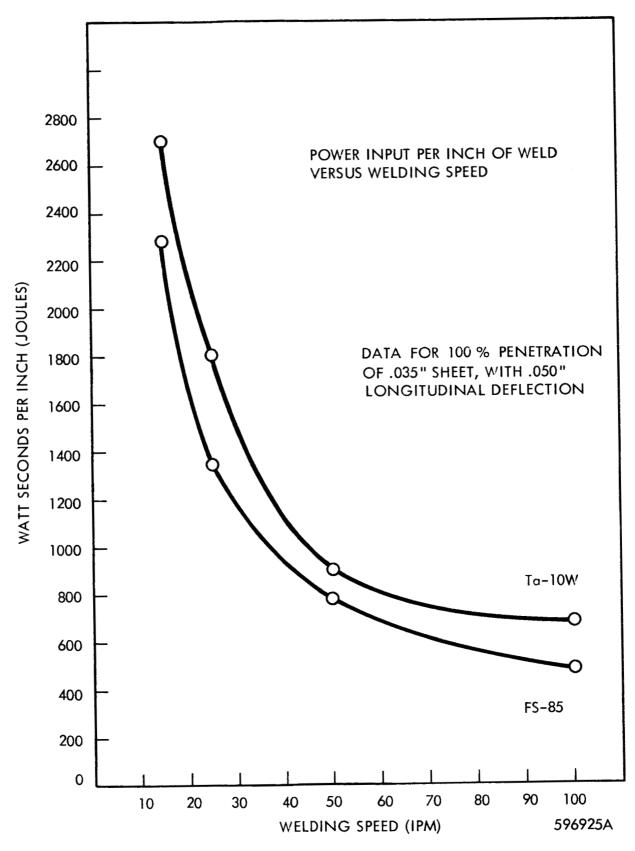
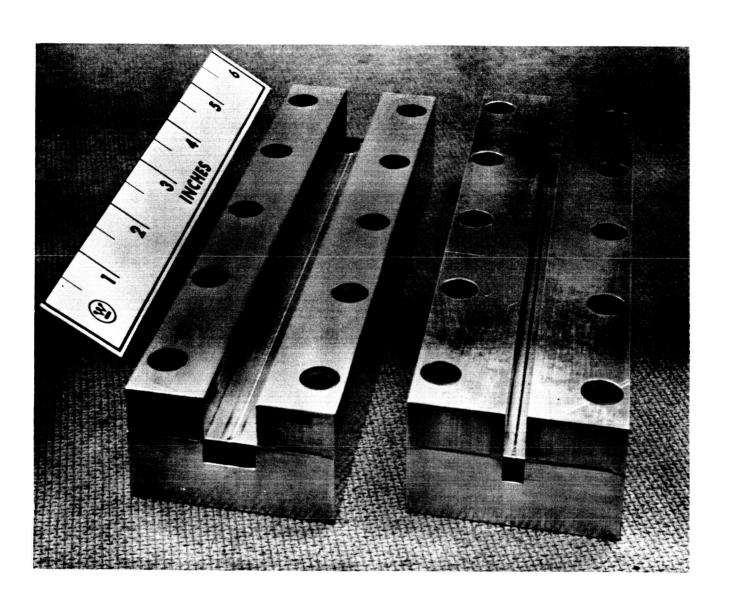


FIGURE 12 - Electron Beam Power Input For Various Weld Speeds





.FIGURE 13 - EB Sheet Butt Weld Clamps



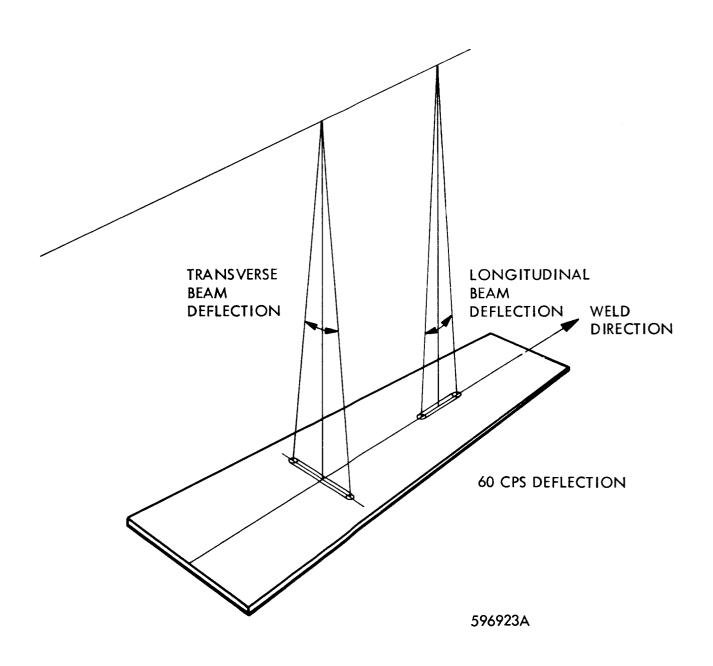


FIGURE 14 - Electron Beam Deflection Pattern



Torr. Present chamber operation includes a bakeout cycle and ultimate pressures after an overnight pumpdown are 1.5×10^{-6} Torr. The addition of a titanium sublimation pump to further reduce the pumpdown time and ultimate operating pressure is contemplated.

c. Bend Testing: Equipment for bend testing was described in an earlier report. The bend test fixture is equipped with a liquid nitrogen cryostat and heater to provide for testing at any temperature from -320°F to +700°F. Testing procedures are fairly straightforward. The bend specimen is bent to an angle of 90°-105° after springback. The bend ductile-brittle transition temperature is identified as the lowest temperature at which a 90° bend can be made without cracking on the tension side of the specimen. Specimens are checked for cracks using both visual and dyepenetrant techniques. Bend test parameters are shown in Figure 15. As defined by this figure, both longitudinal and transverse bend tests are being run in this program. The method of presentation of bend test results used throughout this report is shown in Figure 16.

2. Sheet Weld Evaluation

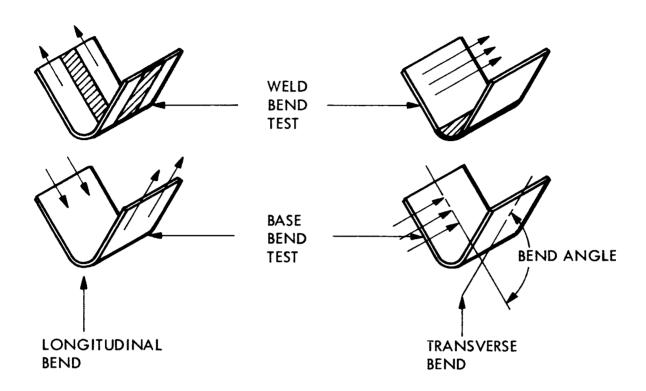
Initial welding studies are directed towards determining the effects of weld process and parameter variations on weldment bend ductile-to-brittle transition temperatures. The weld bend ductile-to-brittle transition temperature is an established weldability criterion for refractory metal alloys, and because of refractory alloy sensitivity to interstitial contaminants, bend testing also provides a measure of weld atmosphere quality.

This initial survey has a dual purpose. Refractory metal alloys generally suffer an increase in ductile-to-brittle transition temperature with welding. A significant increase in transition temperature is naturally undesirable. Hence, the first purpose of the weld parameter investigation is to compare the alloys on the basis of loss of ductility with welding. Weldability in this sense will be defined both by the lowest as-welded bend transition temperature obtainable and also by the transition temperature range for each alloy which is a measure of alloy sensitivity to variations in weld schedules. The second purpose of the weld parameter investigation is to provide data which are sufficiently comprehensive to permit making an intelligent selection of weld parameters for use in the thermal stability studies.

The degree of completion of the weldability phase varies from alloy to alloy. A current summary of bend test data is given in Figure 17. These data will naturally be modified as the program procedes.

Weld bend test results are presented by alloy in Figures 18 through 35 while corresponding welding parameters are presented in Tables 5 through 13. All bend tests were conducted using a 1t bend radius (0.035 inch) except the FS-85 electron beam welds which were tested using a 2t bend radius. The





NOTE: ARROWS SHOW ROLLING DIRECTION

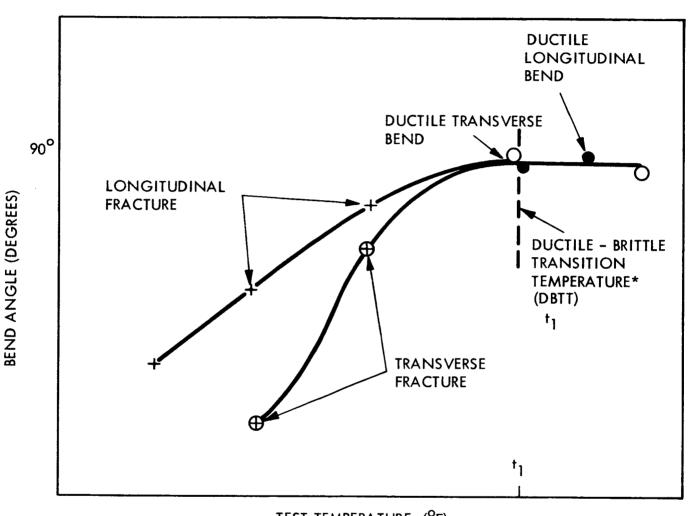
THICKNESS, t = 0.035 INCH
WIDTH = 12t
LENGTH = 24t
TEST SPAN = 15t
PUNCH SPEED = 1 IPM
TEMPERATURE - VARIABLE
PUNCH RADIUS - VARIABLE, GENERALLY 1t, 2t, 4t, or 6t

BEND DUCTILE TO BRITTLE TRANSITION TEMPERATURE = LOWEST TEMPERATURE FOR 90° + BEND WITHOUT CRACKING

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FIGURE 15 - Bend Test Parameters



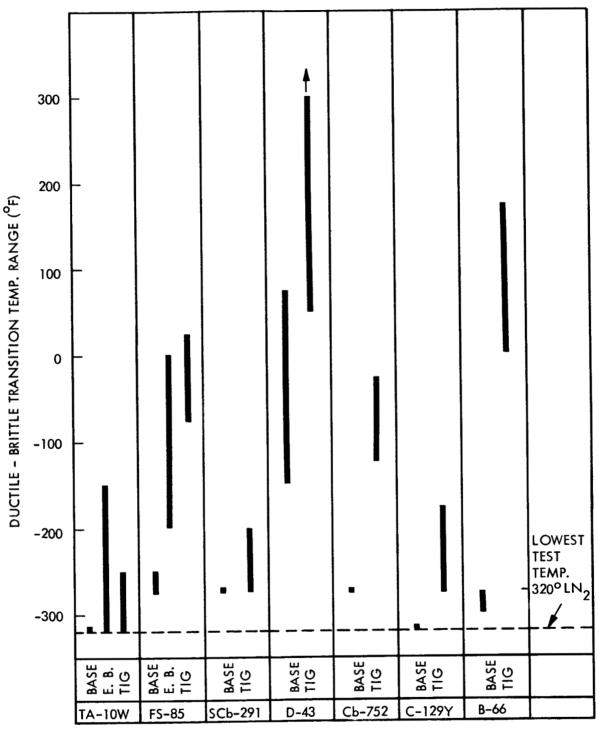


TEST TEMPERATURE (°F)

*TEMPERATURE OF LAST DUCTILE BEND AS CHECKED BY DYE PENETRANT EXAMINATION 596926A

FIGURE 16 - Key For Presentation of Bend Test Data





ALL BENDS 1+ RADIUS EXCEPT FS-85 E. B. WELDS
WHICH ARE 2+ RADIUS 596927A

FIGURE 17 - Summary of Current Bend Test Results



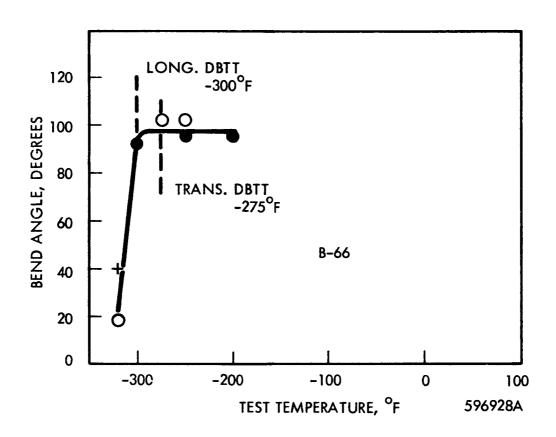


FIGURE 18 - B-66 Base Metal Bend Test Results



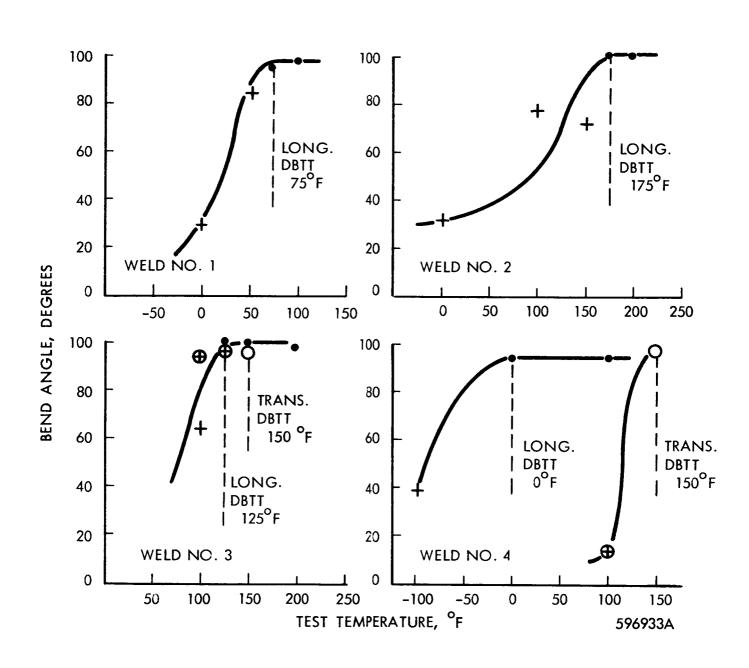


FIGURE 19 - B-66 TIG Weld Bend Test Results



TABLE 5 - B-66 Sheet. TIG Butt Weld Parameters

	G7			Wald Width	Comme	nts
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	D ye Check
1	3/8	15	50	0.11/0.07	Negative	Negative
2	3/8	15	7 0	0.13/0.08	Negative	Negative
3	1/4	15	80	0.14/0.11	1/2" © Starting Crack	Positive
4	1/4	30	90	0.12/0.075	Edge Flash(1)	Negative

Note: Atmosphere monitored (<5 ppm 0_2 and <2 ppm moisture)



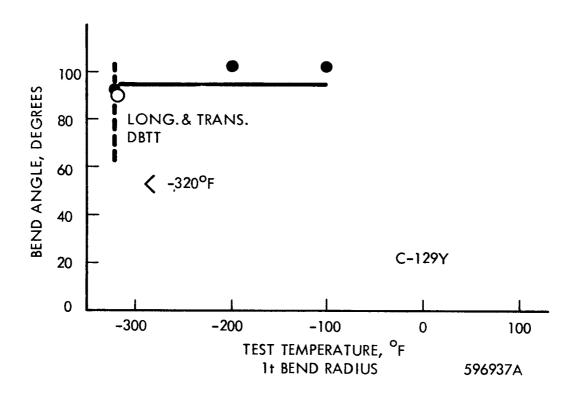


FIGURE 20 - C-129Y Base Metal Bend Test Results



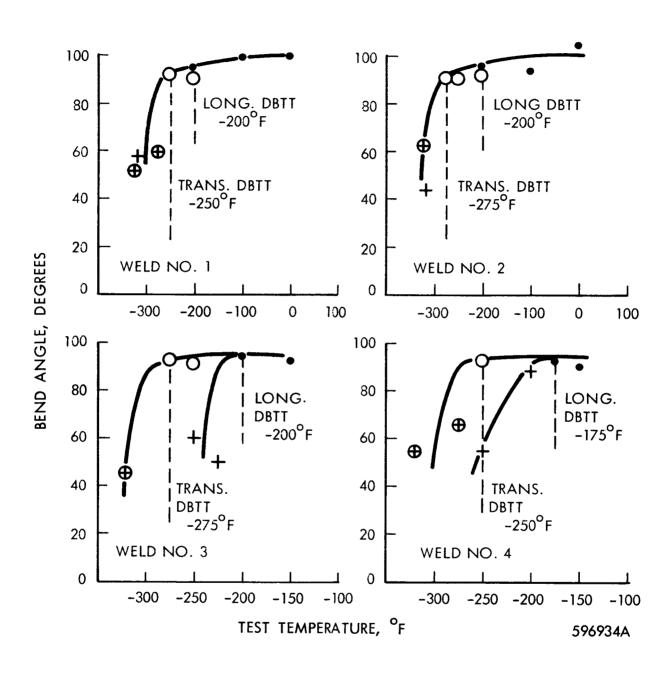


FIGURE 21 - C-129Y TIG Weld Bend Test Results



TABLE 6 - C-129Y Sheet. TIG Butt Weld Parameters

				_	Comme	nts
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	Dye Check
1	3/8	15	70	0.15/0.10	Negative	Negative
2	3/8	30	110	0.18/0.13	Negative	Negative
3	1/4	15	80	0.15/0.11	Edge Flash (1)	Negative
4	1/4	30	102	0.15/0.12	Edge Flash (1)	Negative

Note: Atmosphere monitored (≤ 5 ppm 0_2 and ≤ 3 ppm moisture)



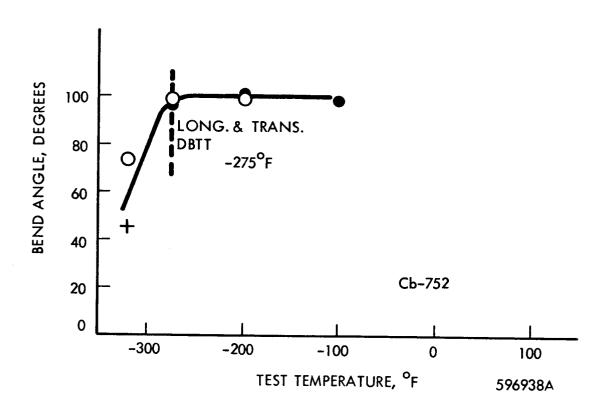


FIGURE 22 - Cb-752 Base Metal Bend Test Results



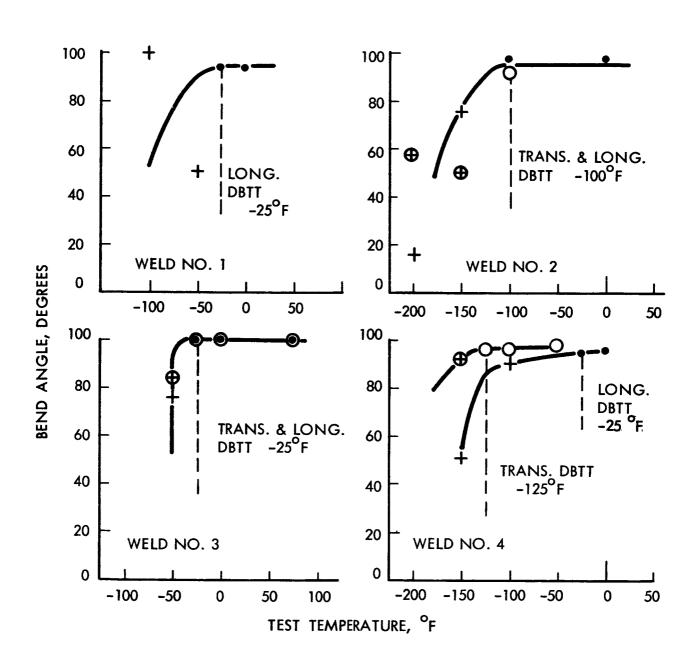


FIGURE 23 - Cb-752 TIG Weld Bend Test Results



TABLE 7 - Cb-752 Sheet. TIG Butt Weld Parameters

					Comment	ts
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	Dye Check
1	3/8	15	70	0.13/0.08	Negative	Negative
2	3/8	30	110	0.16/0.14	1/8" HAZ Crack	1/8" HAZ Crack
3	1/4	15	80	0.16/0.12	Edge Flash(1)	Negative
4	1/4	30	100	0.14/0.10	Edge Flash ⁽¹⁾	Negative

Note: Atmosphere monitored (≤ 5 ppm 0_2 and ≤ 3 ppm moisture)



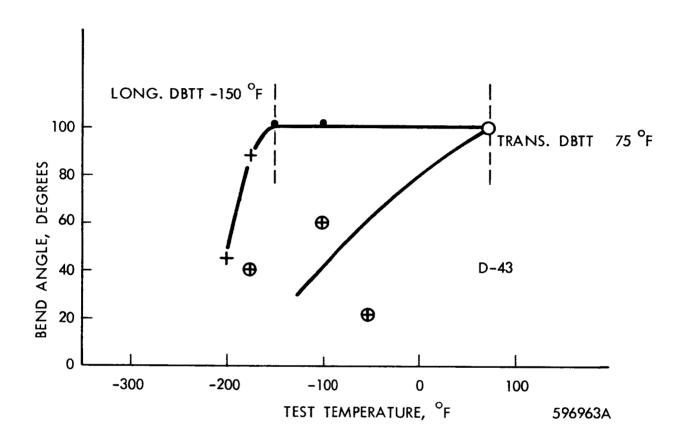


FIGURE 24 - D-43 Base Metal Bend Test Results



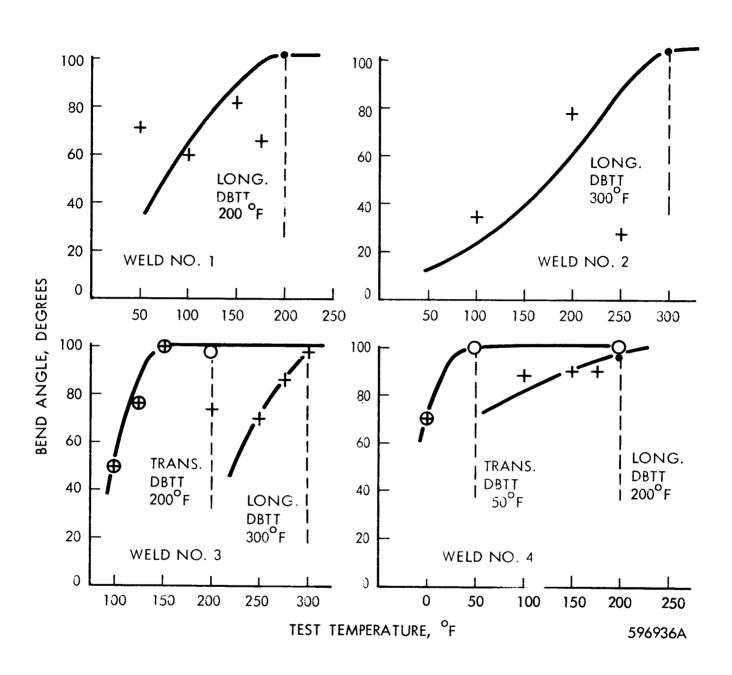


FIGURE 25 - D-43 TIG Weld Bend Test Results



TABLE 8 - D-43 Sheet. TIG Butt Weld Parameters

	G3 -				Comment	3
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	D ye Check
1	3/8	15	60	0.10/0.02	Negative	Negative
2	3/4	15	80	0.18/0.18	Negative	Negative
3	1/4	15	80	0.14/0.12	Edge Flash (1)	Negative
4	1/4	3 0	100	0.14/0.09	Negative	Negative

Note: Atmosphere monitored (<5 ppm 0_2 and <3 ppm moisture)



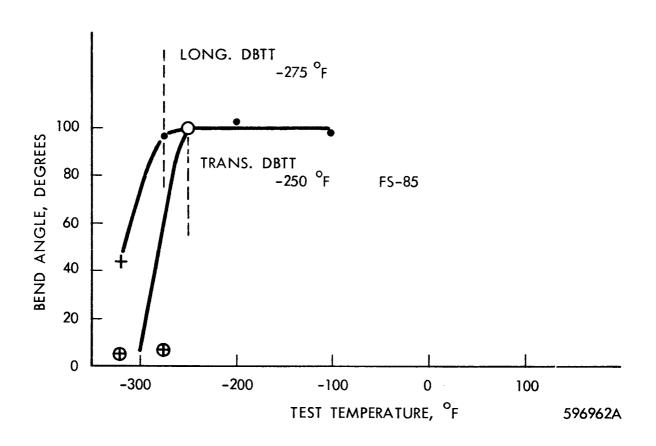


FIGURE 26 - FS-85 Base Metal Bend Test Results



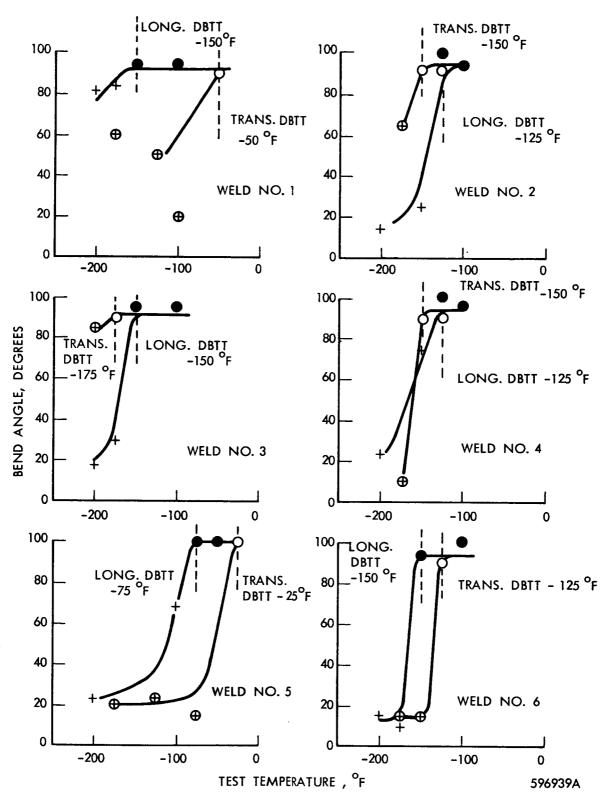


FIGURE 27. - FS-85 EB Weld Bend Test Results



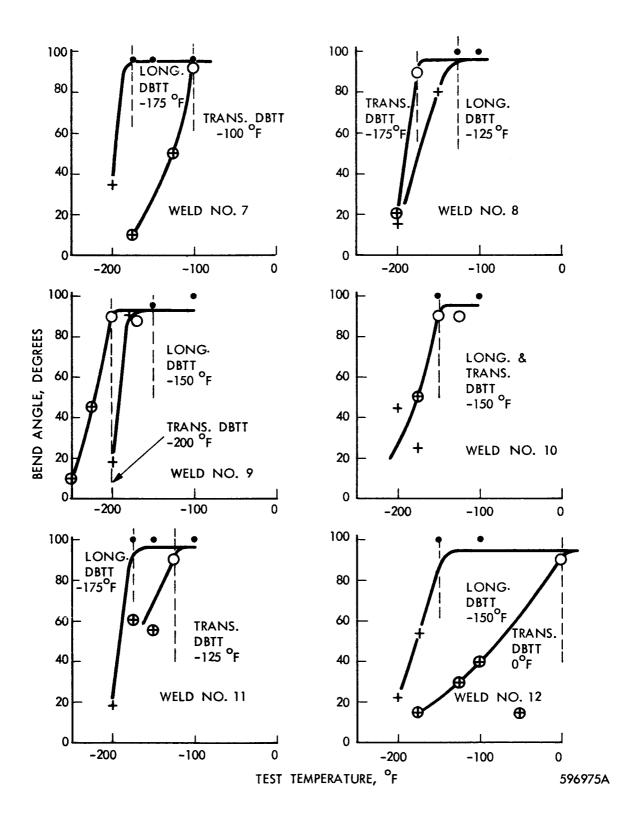


FIGURE 28 - FS-85 EB Weld Bend Test Results



TABLE 9 - Electron Beam Welding Parameters for FS-85

Vacilim2	(Torr)	6 × 10 ⁻⁶	6 × 10 ⁻⁶	6 × 10 ⁻⁶	9-01 × 9	6 × 10 ⁻⁶	6 × 10-6	6 × 10 ⁻⁶	6 x 10 ⁻⁶	6 × 10-6	6 × 10 ⁻⁶	6 × 10 ⁻⁶	6 × 10 ⁻⁶
Weld Bead Width (inches)	Bottom	0.020	0.027	0.027	0.037	0,040	090.0	0.020	0.025	0.026	0.027	0.027	0.050
Weld Bead Winches	Top	0.027	0.038	0.045	0.047	0.049	0.070	0.027	0.036	0.038	0,040	0.038	090.0
Watt-Sec.	per inch	720	495	266	1370	2280	2280	720	567	266	1370	2280	. 2280
Power	(watts)	750	825	099	570	570	570	750	825	099	570	570	570
Chill Spacing	(inches)	0.250					\rightarrow	760.0					>
Current	- 1	5.0	5.5	7.7	3.8	3.8	3.8	5.0	5.5	7.7	3.8	3.8	3.8
Deflection1	(inches)		I-0.050"	I-0.050"	I-0.050"	I-0.050"	T-0.050"		I-0.050"	I-0.050"	L-0.050"	I-0.050"	T-0.050"
	(ipm)	100	100	20	25	15	15	100	100	50	25	15	15
Weld	No.	Н	R	σ.	7	2.	9	7	∞	6	10	7	12

All Welds Made at 150 KV, Optical Focusing L is longitudinal T is transverse See Figure 14 i

Current evacuation practice provides pressures of 1.5 x 10-6 Torr 8



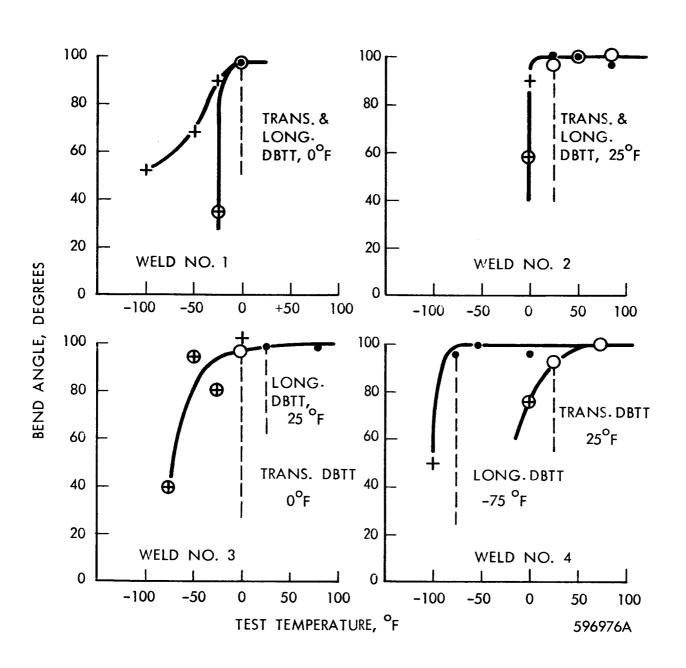


FIGURE 29 - FS-85 TIG Weld Bend Test Results



TABLE 10 - FS-85 Sheet. TIG Butt Weld Parameters

					Comments	
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	D ye Check
1	3/8	15	70	0.14/0.11	Negative	Negative
2	3/8	30	110	0.17/0.15	Negative	1/16" HAZ Crack
3	1/4	15	85	0.15/0.14	Edge Flash ⁽¹⁾	Negative
4	1/4	30	104	0.14/0.11	Edge Flash ⁽¹⁾	Negative

Note: Atmosphere monitored (<5 ppm 0_2 and <3 ppm moisture)



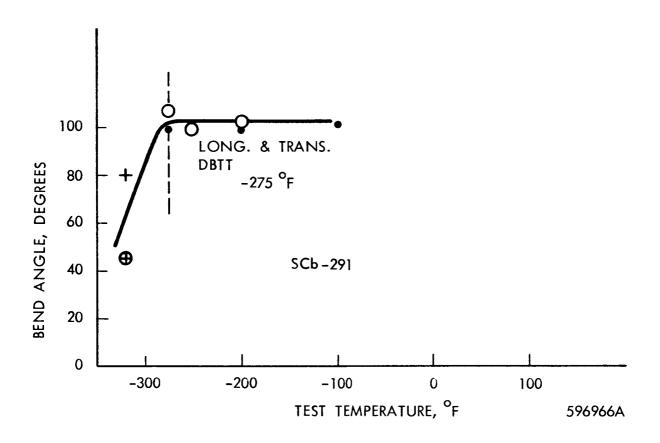


FIGURE 30 - SCb-291 Base Metal Bend Test Results



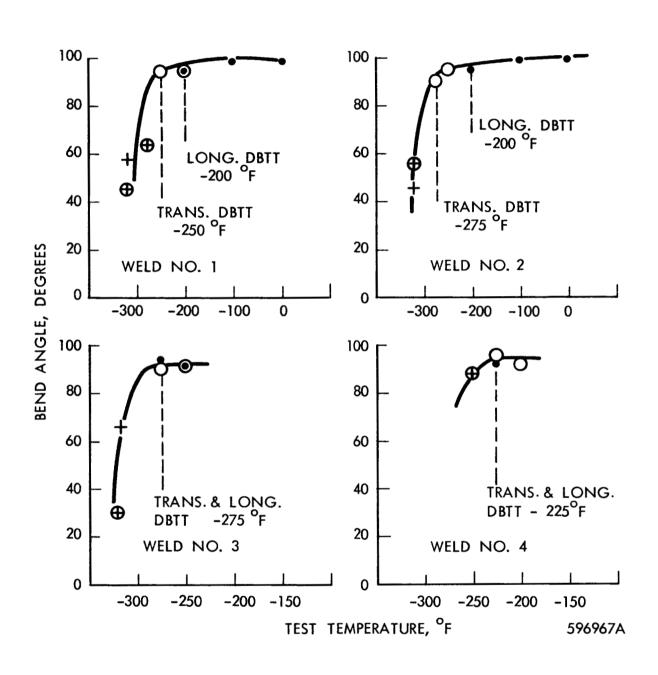


FIGURE 31 - SCb-291 TIG Weld Bend Test Results



TABLE 11 - SCb-291 Sheet. TIG Butt Weld Parameters

					Commen	ts
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	D ye Check
1	3/8	15	70	0.15/0.13	Negative	Negative
2	3/8	30	115	0.18/0.16	1/8" HAZ Crack	1/8" HAZ Crack
3	1/4	15	83	0.16/0.15	Edge Flash (1)	Negative
4	1/4	30	103	0.13/0.11	Edge Flash (1)	Negative

Note: Atmosphere monitored (\leq 5 ppm 0₂ and \leq 3 ppm moisture)



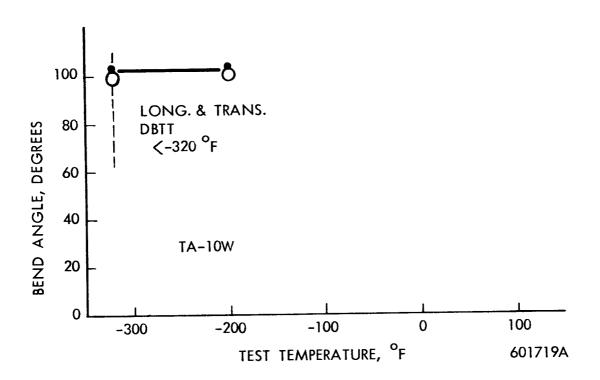


FIGURE 32 - Ta-10W Base Metal Bend Test Results



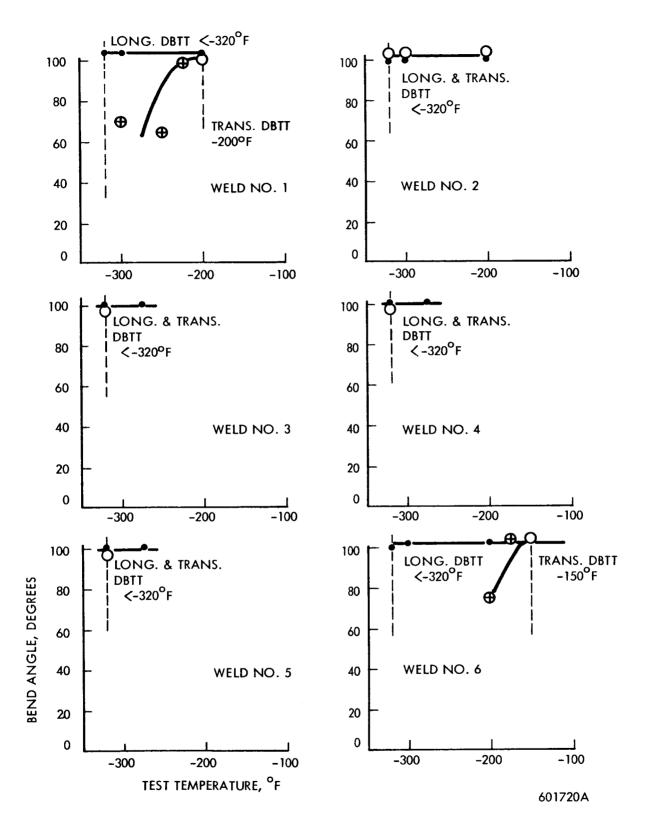


FIGURE 33 - Ta-10W EB Weld Bend Test Results



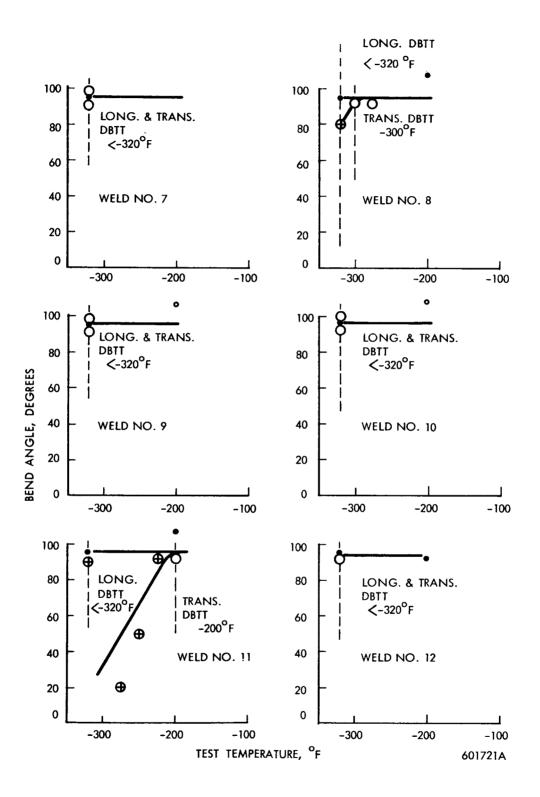


FIGURE 34 - Ta-10W EB Weld Bend Test Results



TABLE 12 - Electron Beam Welding Parameters for Ta-lOW

Vacuum ²	(Torr)	9×10-6	9×10-6	1.9 × 10-6	1.4 × 10 ⁻⁶	1.4 x 10 ⁻⁶	2 x 10 ⁻⁵	1 × 10 ⁻⁵	1 × 10 ⁻⁵	1 x 10 ⁻⁵	8 x 10-6	5 x 10-6	5 × 10 ⁻⁶
d Bead Width (inches)	Bottom	0.015	0.020	0,040	0.055	0.050	090.0	0.010	0.030	0.020	0.025	0.032	0.062
Weld Be	Top	0.030	0.025	0.055	0,060	0.065	0.070	0.020	0.030	0,040	870.0	0,040	0.070
Watt-Sec.	per inch	240	089	006	1800	2700	2400	240	089	066	1980	3300	2960
Power	(watts)	006	1130	750	750	675	009	006	1130	825	825	825	07/
Chill Spacing	(inches)	0.250				\rightarrow	0.250	760.0				\rightarrow	760.0
Current	(ma)	9.9	8.2	5.0	5.0	4.5	7-7	9.9	8.2	5.5	5.5	5.5	6.4
Deflection1			L-0.050"	L-0.050"	L-0.050"	I-0.050"	T-0.050"		I-0.050"	L-0.050"	I-0.050"	L-0.050"	T-0.050"
Speed	(ipm)	100	100	50	25	15	15	100	100	55	25	15	15
Weld	No.	Н	æ	~	7	2	9	7	∞	6	10	11	12

l. L is longitudinal
 T is transverse
 See Figure 14

Current evacuation practice provides pressures of 1.5 \times 10-6 Torr ς,



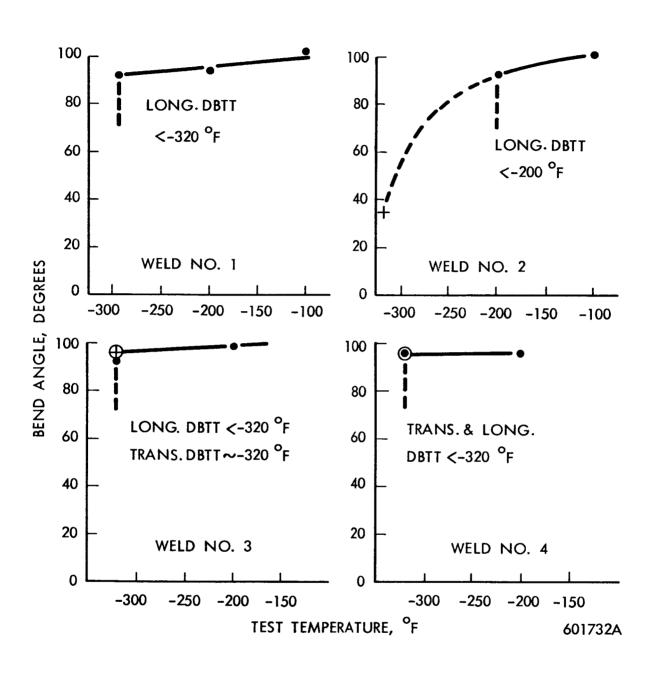


FIGURE 35 - Ta-10W TIG Weld Bend Test Results



TABLE 13 - Ta-10W Sheet. TIG Butt Weld Parameters

					Comme	nts
Weld No.	Clamp Spacing (Inch)	Speed (ipm)	Current Amperes	Weld Width Top/Bottom (Inch)	Visual Inspection	D y e Check
1	3/8	15	105	0.18/0.18	Negative	Negative
2	3/4	15	90	0.16/0.15	Negative	Negative
3	1/4	15	90	0.14/0.08	Negative	Negative
4	1/4	30	126	0.13/0.08	Edge Flash (1)	Negative

Note: Atmosphere monitored (<5 ppm 0_2 and <3 ppm moisture)



data are presented without comment. Discussion and interpretation of results will be deferred until a more complete and comprehensive analysis is possible.

In the tabulated weld parameter data for TIG welds frequent edge flashing was noted when using narrow clamp spacing (1/4 inch). This apparently resulted from occasional arc sticking on the copper chill bars. In future welds the copper inserts will be replaced with molybdenum to permit narrow weld clamping.

3. Weld Restraint Tests

Two types of weld restraint tests are being made on each alloy. A circular groove restraint test is being used for plate material. A bead-on-plate patch test is being used for the sheet material. Drawings of the test specimens were presented in a previous report. 1

Weld restraint tests were completed for both plate and sheet of seven alloys. Results of these tests are summarized in Table 14. All patch tests were examined visually at 30x and inspected by radiography and dyepenetrant testing. All welds had indications at weld end craters. These indications were not listed as "positive" since they could have been eliminated by weld current tailing. B-66 had a positive indication of a 1/8 inch weld start crack in both radiographic and dye-penetrant testing. Positive indications in FS-85 radiographs were traced to weld texture, not defects. Considerable distortion, indicating the severity of weld stresses, was noted on all samples. Distortion measurements were made and are listed in Table 14. There appears to be little difference in weld distortion among these alloys. The FS-85 and B-66 patch tests are shown in Figure 36. Other specimens were included in the Second Quarterly Report.²

The circular groove plate weld data are also shown in Table 14. All specimens welded satisfactorily. The completed welds are shown in Figures 37 through 40. These specimens were welded with a fusion root pass (to increase the effective weld depth) and two additional passes using filler metal.

C. TIG WELD ATMOSPHERE MONITORING

1. Monitoring System Performance

High moisture levels previously reported were eliminated by removing the Moisture Monitor from the manifold and installing it directly to the welding chamber. This eliminated the apparent moisture contamination from the common sampling pump and manifold system and improved response. The total length of the 0.055 inch I.D. stainless steel sampling line with the meter connected in this manner is 36 inches. Brazed or soldered connections were not



TABLE 14 - Restraint Test Summary

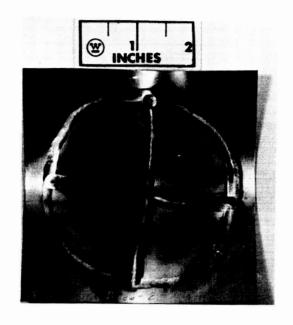
	Be	Bead-on-Plat	e Patch T	-on-Plate Patch Test (Sheet)			Circu	Circular Groove (Plate)	e (Plate)
		Weld	Dye		Distortion	no		Dye	Distortion
Alloy	Visual	Width	Check	X-Ray	Angle (Max)	Inches ¹	Visual	Check	(inches)
B-66	Neg.	0.23	Neg.	Positive ²	31°	69.0	Neg.	Neg.	70.0
C-129Y	Neg.		Neg.	Neg.	30°	0.63	Neg.	Neg.	70.0
Cb-752	Neg.	0.34	Neg.	Neg.	28°	0.73	Neg.	Neg.	0.03
D-43	Neg.	0.23	Neg.	Neg.	32°	09.0	Neg.	Neg.	0.02
FS-85	Neg.	0.15	Neg.	Positive ³	36°	0.76	Neg.	Neg.	0.02
SCb-291	Neg.	0.20	Neg.	Neg.	32°	69.0	Neg.	Neg.	. 0.03
Ta-10W	Neg.	0.17	Neg.	Neg.	30°	0.70	Neg.	Neg.	70.0

Closest distance between two parallel planes on opposite sides of weldment.

3. Positive x-ray indication not identified in consequent examination.

^{2. 1/8} inch starting crack identified on one leg of weld.





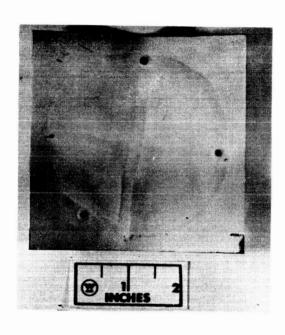


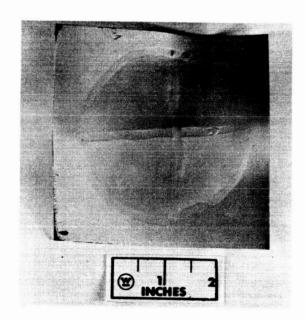
53564 B-66

As Welded

53564

FS-85





53564

Dye Penetrant Inspected

53564

B-66 FS-85

FIGURE 36 - FS-85 and B-66 Bead-on-Plate Patch Tests







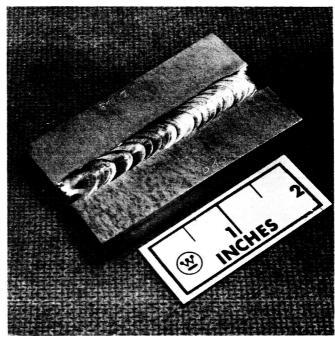
43658 126–3

FIGURE 37 - Ta-10W Circular Groove Restraint Test and Sample Butt Welds



D-43

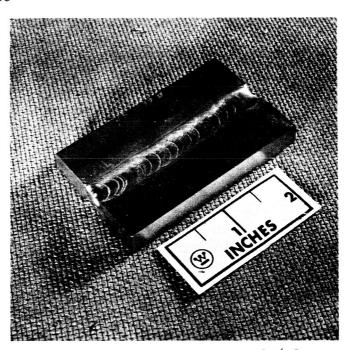




126 - 5

B-66



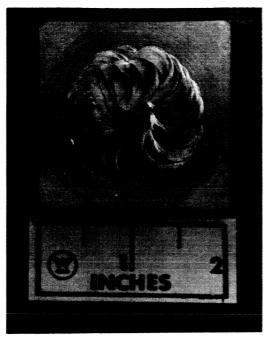


156-4

FIGURE 38 - D-43 and B-66 Circular Groove Restraint Tests and Sample Butt Welds



Cb-752

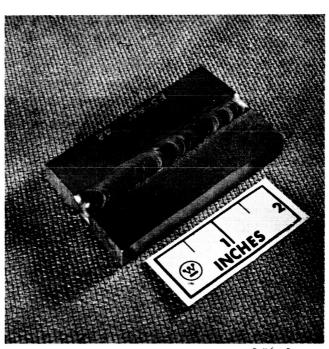




126-1

FS-85



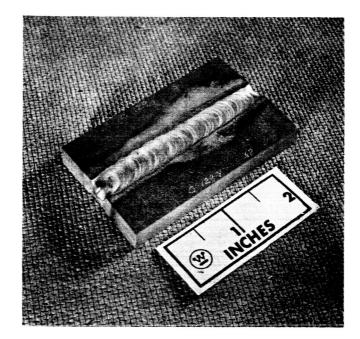


156-5 156-3

FIGURE 39 - Cb-752 and FS-85 Circular Groove Restraint Tests and Sample Butt Welds

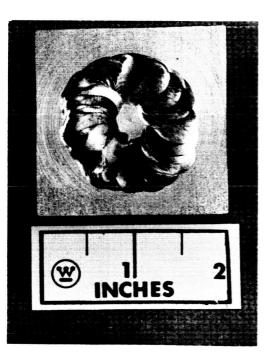


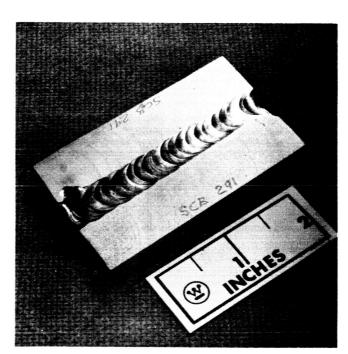




156-6

SCb-291





126-2

FIGURE 40 - C-129Y and SCb-29l Circular Groove Restraint Tests and Sample Butt Welds



used. Following this change, readings of zero water content were obtained immediately after backfilling, remaining below 1 ppm for 1 to 2 hours. All moisture monitor data described in this report were obtained with this modification.

2. Weld Chamber Glove Tests

Three types of gloves, polyvinyl chloride, neoprene, and butyl rubber, were functionally tested for oxygen and water vapor permeability. The chamber was normally evacuated and backfilled with helium, and oxygen and water vapor levels were monitored for periods up to 24 hours.

Figure 41 shows the increase in chamber atmosphere oxygen with time. The data available to date indicate that glove composition has little effect on oxygen contamination of chamber atmosphere since all gloves showed a significant contamination rate over the sealed, without gloves, chamber. The gradual increase in oxygen level apparently is related to the glove permeability since the ultimate vacuum prior to backfilling does not have a significant effect on the rate of contamination.

Figure 42 shows the change in chamber water vapor level with time. Of immediate interest is the significant relationship of ultimate chamber vacuum to contamination rate. Two greatly different contamination rates were obtained for neoprene gloves, the one with a lower rate having been evacuated, backfilled with helium, and evacuated again to 1.5×10^{-5} Torr. The evacuation procedure for the high contamination rate consisted of a single evacuation cycle to 2.5×10^{-5} Torr including a heat lamp chamber bakeout.

Significantly lower water vapor contamination rates have been obtained following evacuation to lower ultimate vacuum. Although the exact relationship is not known, the following effects are thought to occur:

- 1. The lower pressures remove more sorbed water vapor from the chamber, gloves, and tooling and outgassing of the resultant cleaner surfaces following backfilling is decreased.
- 2. Pumping to lower pressures removes more sorbed water vapor as above, and the cleaner surfaces have a substantial capacity to resorb water vapor either brought in with the initial charge of helium or gradually diffused through the gloves and seals. A significant sorption or pumping action seems to occur since lower water vapor measurements have been obtained from the backfilled chamber (zero ppm) than those obtained directly from a helium bottle (2.5 ppm).



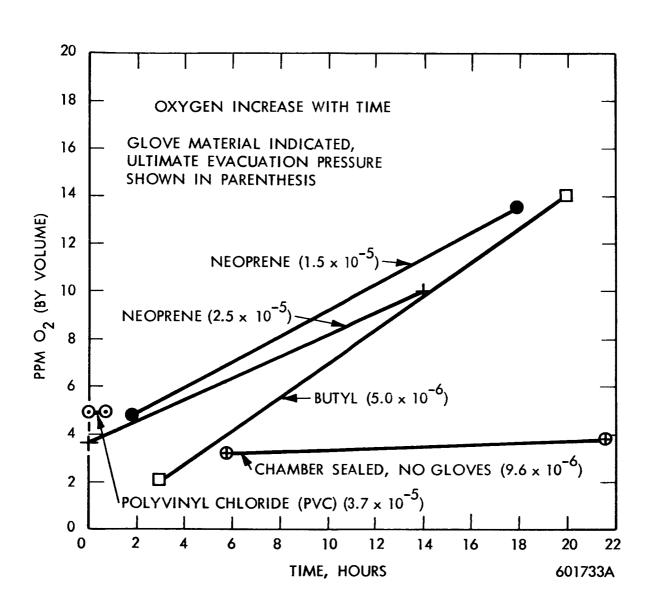


FIGURE 41 - Atmosphere Decay Curve Measuring Oxygen



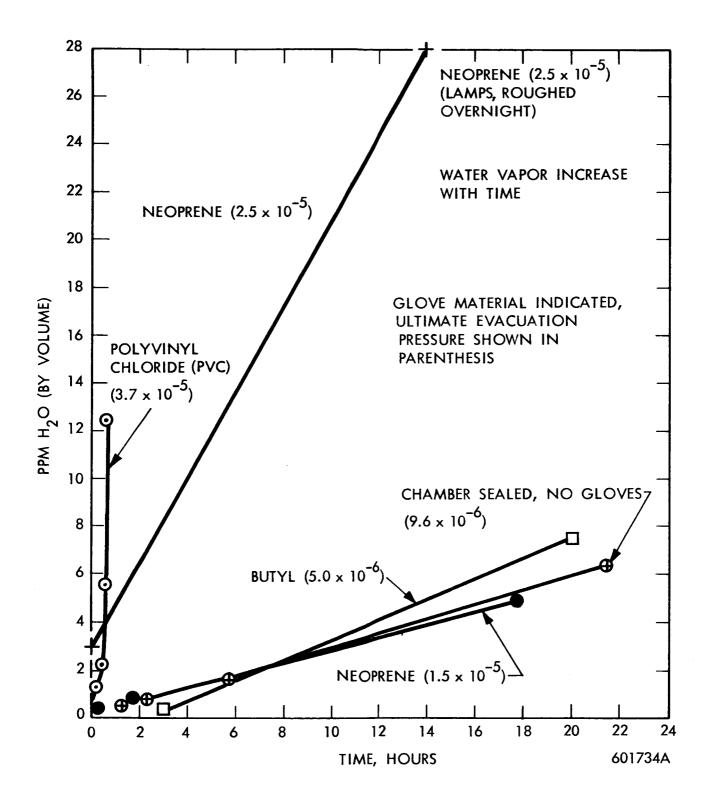


FIGURE 42 - Atmosphere Decay Curve Measuring Moisture



The following general observations on glove behavior were made:

Neoprene: Neoprene gloves behaved satisfactorily under a vacuum pressure of 6 x 10^{-6} Torr. Permeability to oxygen and water vapor was low enough to allow up to 8 hours of welding time with the oxygen and water vapor levels below 5 ppm. The electrical insulating properties of neoprene were compatible with manual TIG welding using a high frequency arc start.

Severe blackening of copper tooling was observed during the chamber evacuation cycle. Spectrographic examination indicated the contaminant was sulfur which apparently outgases from the neoprene gloves at low pressures and reacts with the copper to form sulfides. Since observation showed the contamination spread in a line of sight path from the gloves during evacuation, all tooling and specimens were wrapped in aluminum foil until the chamber was backfilled. With this precaution no visual contamination of the copper tooling occurred.

Polyvinyl Chloride: PVC gloves released a considerable amount of oily substance during evacuation which coated the interior surfaces of the chamber and sight port. The oil evolution was observed on two separate tests of PVC gloves and was totally absent from identical tests of neoprene and butyl rubber gloves. The oil is probably a plasticizer used in the PVC glove manufacturing process. The present evacuation procedure includes a heat lamp chamber bakeout and the higher glove temperature may aggravate glove volatization problems. The ultimate vacuum obtainable during the evacuation cycle was noticeably poorer when using the PVC gloves, apparently being limited by the plasticizer vapor pressure. PVC gloves will not be used in the welding program for this reason.

Butyl Rubber: One pair of butyl rubber gloves was evaluated. These demonstrated good vacuum stability and gas permeability equal to neoprene. Unusual outgassing under vacuum was not observed. However, the particular type tested had poor dielectric properties and were unsuitable for welding. Other types of butyl rubber gloves will probably be tested in the future.

3. Helium Purification System

The use of a recirculating purification system is being considered for TIG welding. Evaluation of an adsorbent bed helium purifier for use as either a chamber atmosphere recirculator or supply purifier was initiated. The system, manufactured by Engelhard Industries, is equipped with a self-contained recirculator pump providing a system recirculating capacity of 600 scfh. Liquid nitrogen cooled charcoal and molecular sieve columns are designed



to remove impurities to 1 ppm and dual purification columns allow automatic vacuum bakeout regeneration for continuous operation.

A short time recirculation-purification test was run and the performance is shown on Figure 43. The chamber was evacuated, backfilled with helium, and the purged recirculator was turned on. The initial high impurity level is apparently caused by residual gas in the purifier and a continuously operated purifier would avoid this problem. After one hour of operation, a gas sample of the chamber atmosphere was obtained for which the mass spectrometer analysis is shown in Table 15. All impurity levels were comparable to a normal backfilled chamber analysis with the exception of nitrogen which is nearly twice as high as expected, indicating the purifier was not removing nitrogen as effectively as oxygen and water vapor. Future tests will be run over a longer period of time allowing optimizing adjustments of regeneration time and temperature to be made.

D. EQUIPMENT CHECKOUT

1. Ultra High Vacuum Aging Furnaces

Four aging furnaces have been delivered and are presently being installed. A completed unit is shown in Figure 44. These units are designed for ultra high vacuum performance. They are bakeable to 250°C and employ no organic materials for high vacuum pumping or sealing. A 500 l/sec. sputter ion pump is used for high vacuum pumping. Roughing is accomplished by connecting a sorption pump to the bakeable roughing valve. Provisions have been made for attaching a titanium sublimation pump to accommodate high gas loads.

The furnace is located in the throat of the sputter ion pump providing maximum pumping efficiency. A four inch diameter by ten inch long split tube tantalum resistance heater is being used. The inner radiation shields are also of tantalum while the outer shield is a water cooled copper jacket.

Temperature control can be accomplished by sensing with either a furnace thermocouple or thermal converter. With thermal converter control, power input is held constant and control response is independent of the furnace thermal inertia and is consequently faster than possible using thermocouple control. Furnace temperature stability was demonstrated by operating with thermal converter control under varying line voltage conditions, Figure 45, and with varying cooling water temperature, Figure 46. The thermal converter control proved adequate for both types of ambient fluctuation.

During factory checkout a pressure of 1.4 \times 10⁻⁹ Torr was obtained



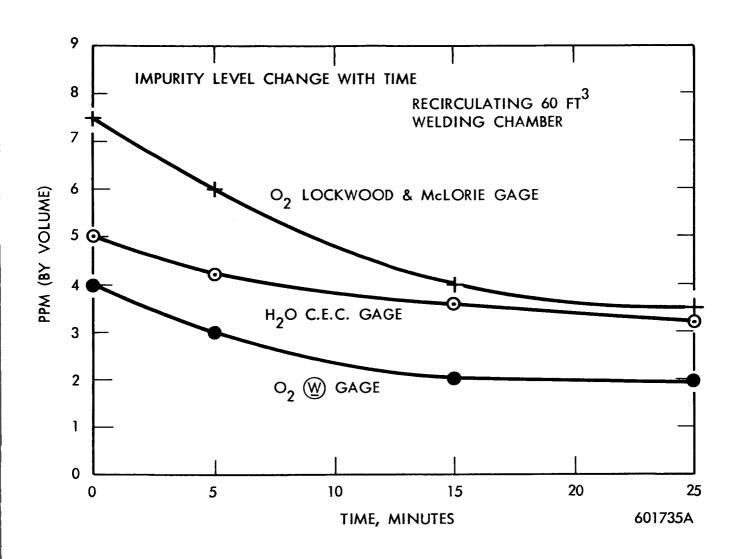


FIGURE 43 - Helium Purifier Performance



TABLE 15 - Mass Spectrometer Analysis of Purified Helium

Element		ppm	
N ₂		19.0*	
Chlorinated Hydrocarbon		7.8	
Ne		5.3	
02		2.7	
co ₂		0.9	
A		0.5	
н ₂		0.4	
н ₂ 0		0.0	
	Total	36.6	

^{*} Normal 4:1 ratio would be 10.8 ppm



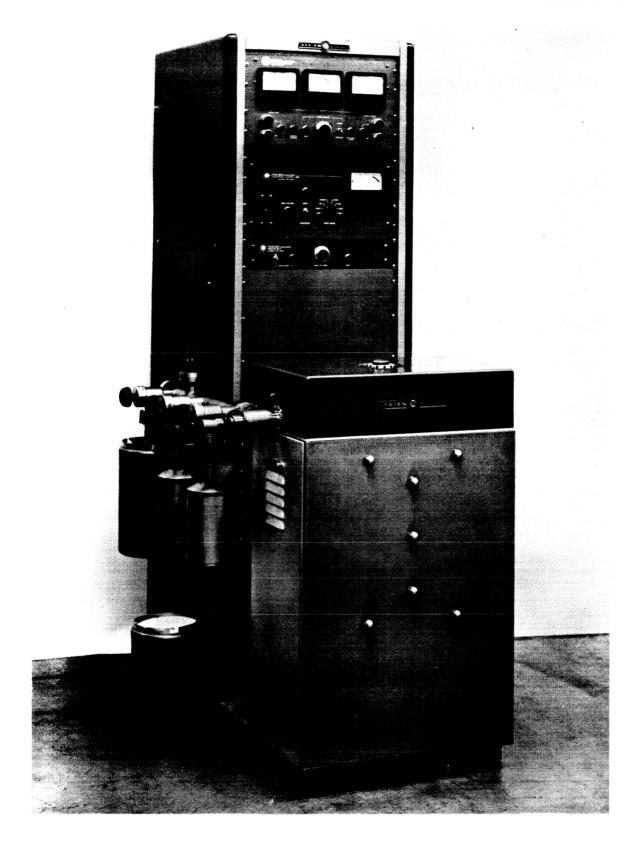
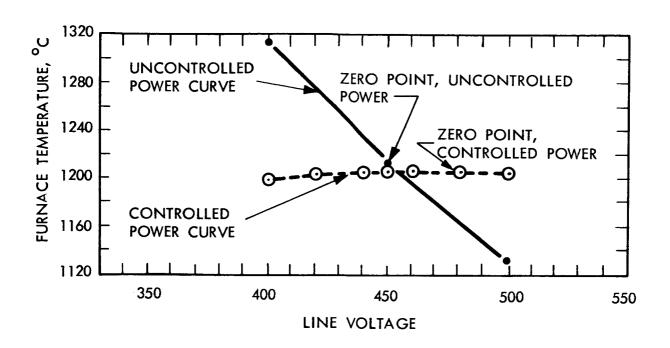


FIGURE 44 - Ultra High Vacuum Aging Furnace





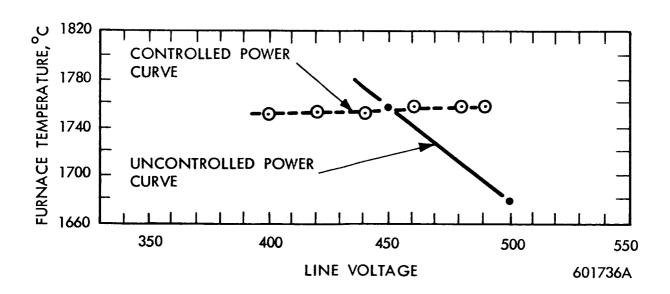


FIGURE 45 - Effect of Line Voltage Variation on Aging Furnace Temperature



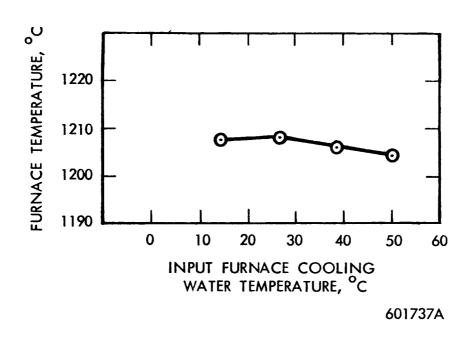


FIGURE 46 - Effect of Furnace Cooling Water
Temperature on Furnace Temperature



at 2500°F following bakeout at 480°F with sputter ion pumping. With the addition of titanium sublimation pumping a pressure of less than 6 x 10^{-10} Torr was obtained at 3270°F. Pressure measurements were made with nude ion gages. Check gage calibration tests at these pressures demonstrated that somewhat lower readings, 8 x 10^{-10} Torr and 2.5 x 10^{-10} Torr respectively, are obtained using a Keisman gage.

2. Weld Contamination Evaluation

An effort was made to determine the relative weld contamination of helium atmosphere TIG welding and electron beam welding at various chamber pressures.

Two measurements of weld contamination were used: chemical analysis of weld metal for interstitial content and weld bend ductile-brittle transition temperature. Pure Cb and SCb-291 were used to measure interstitial contamination and ductile-brittle behavior respectively. The solid solution strengthened SCb-291 (Cb-10W-10Ta) was used to minimize the unpredictable ductile-brittle transition effects observed in alloys containing reactive elements (Zr or Hf) which tend to "getter" the interstitial contaminants. Table 16 outlines the evaluation program. To obtain sufficient weld metal for nitrogen and oxygen analyses, six over-lapping electron beam passes were made to produce a total bead width equal to the 0.250 inch TIG pass. Complete weld parameters are listed in Table 17.

Cross sections of the pure columbium welds were examined for hardness variation as shown in Figure 47. Recrystallization treatment prior to welding produced a coarse grained base metal structure that was impossible to distinguish from the heat affected zone.

The TIG weld cross section exhibited slightly higher hardness values than the EB welds. The measured base metal oxygen content of 150 ppm corresponds to a Vickers hardness value of 80.3 The measured base metal hardness was 75 to 80 DPH.

Table 18 lists the measured oxygen and nitrogen levels of the columbium metal for the three electron beam welding conditions. Samples for O_2 and N_2 analysis were sectioned entirely from the weld metal. Oxygen content was obtained by vacuum fusion analysis and nitrogen was measured by the Kjeldahl method.

No significant change in either nitrogen or oxygen content was observed in the three EB welds. The indicated nitrogen content was slightly higher in all three than in the base metal and oxygen levels were apparently reduced although the data is all within the range of analysis reproducibility.



TABLE 16 - Weld Contamination Evaluation Program

	Specimen Identification		
Welding Conditions	Ср	SCb-291 (Cb-1 OW- 10Ta)	
TIG Weld	Cb-TIG	SCb-TIG	
EB Welds			
1 x 10 ⁻³ Torr 1 x 10 ⁻⁴ Torr 1 x 10 ⁻⁵ Torr	Cb-3 Cb-4 Cb-5	SCb-3 SCb-4 SCb-5	

TABLE 17 - Parameters for Weld Contamination Tests

TIG Welding Parameters	EB Weld Parameters	
0.060" electrode	100 K.V.	
0.020" arc gap	5 M.A.	
16 volts	0.050" transverse deflection	
80 amps	25 ipm	
15 ipm	1200 Joules/inch	
5120 Joules/inch	Bead Width	
0.230"/0.230" bead width	0.060"-0.050" @ 10 ⁻³ Torr	
Chamber Atmosphere	0.050"-0.050" @ 10 ⁻⁴ Torr	
Cb - 2.5ppm 0 ₂	0.050"-0.040" @ 10 ⁻⁵ Torr	
SCb-291 - 2.5ppm 0 ₂		



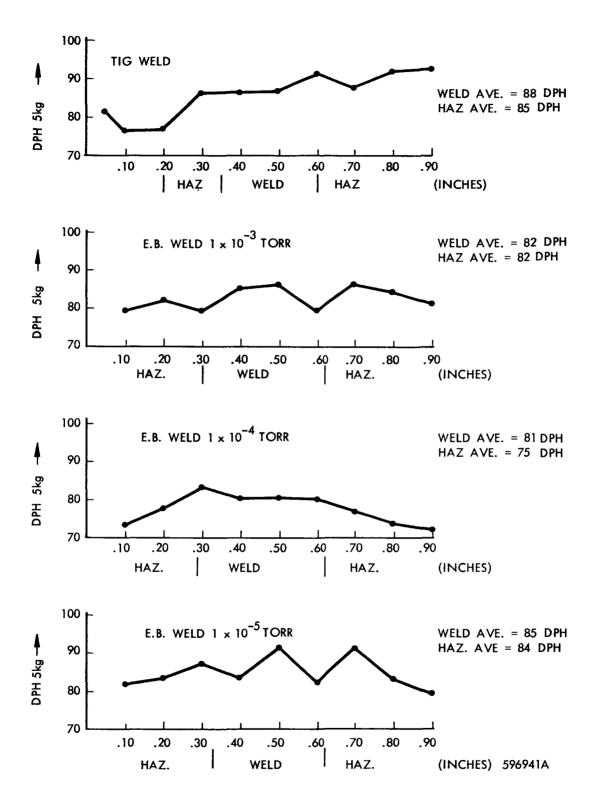


FIGURE 47 - Hardness Traverses of Columbium Welds



TABLE 18 - Nitrogen and Oxygen Analysis of Columbium Welds

Weld	O ₂	N ₂ ppm
Base Metal	150	40
EB 1 x 10 ⁻³ Torr	120	50
EB 1 x 10 ⁻⁴ Torr	150	60
EB 1 x 10 ⁻⁵ Torr	140	50



As a further comparison of the relative contamination and embrittlement characteristics of TIG welds and EB welds at various chamber pressures, longitudinal and transverse bend tests were obtained for typical welds in SCb-291. A comparison of the bend test results, Figure 48, shows no significant difference between the EB welds at three chamber pressures, but the longitudinal bend test of the TIG weld does show a higher transition temperature. In all but one of the transverse brittle bends, the fracture occurred in the heat affected zone indicating this area to be less ductile than the weld. This tends to explain the behavior of the TIG weld bend tests which exhibit ductile transverse bending to liquid nitrogen temperature (-320°F) since the large weld bead size (0.200 inch) permits most of the straining to occur in the weld metal. The longitudinal bend, which equally strains the weld metal and heat affected zone, initiated fractures in the heat affected zone at higher temperatures.

In general, these tests demonstrated that electron beam welds of Cb and SCb-29l made with different chamber pressures have no significant difference in weld bend behavior or oxygen and nitrogen contamination. The SCb-29l TIG weld bend behavior differed from the EB welds in that the transverse bends were ductile to -320°F while longitudinal bend testing resulted in a 75°F increase in transition temperature. Since no difference was noted in EB weld ductility and transverse TIG welds were ductile to -320°F, it appears that weld atmosphere purity per se, over the range evaluated, did not affect the bend transition temperature. Oxygen and nitrogen analyses of TIG welds made in pure columbium and selected program alloys are in process.



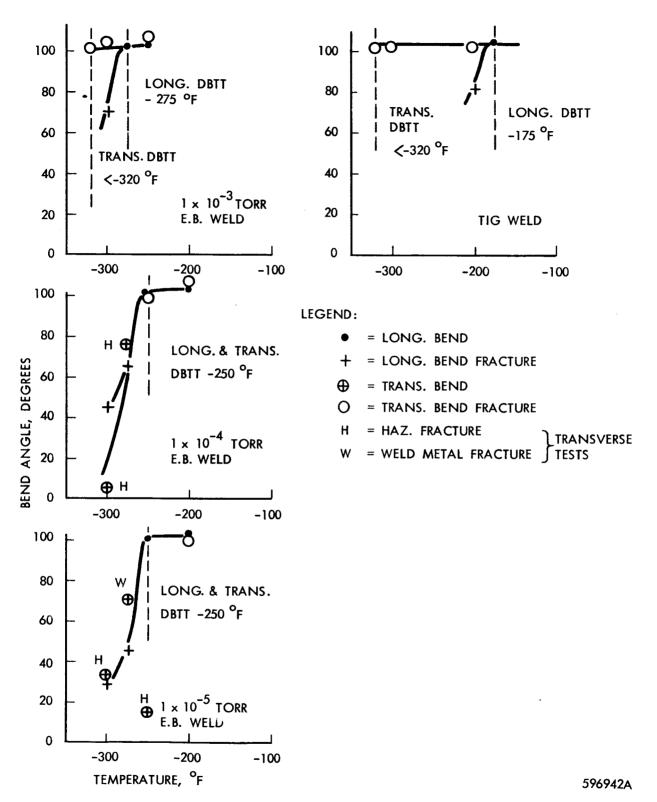


FIGURE 48 - Effect of EB Vacuum on Bend Transition Temperature of SCb-291



IV. FUTURE WORK

Weld parameter optimization studies will continue for the seven alloys discussed in this report. Welding studies will be initiated in W-25 Re alloy for which delivery is now complete and also AS-55 and T-222 should these become available. Upon the selective completion of the weld parameter evaluation, alloys will be screened for satisfactory post weld anneals.

Fabrication and evaluation of plate weldments will also be initiated.

Checkout of the aging furnaces should be complete and ready for thermal stability screening of the alloys.



V. REFERENCES

- 1. G. G. Lessmann and D. R. Stoner, "Determination of the Weldability and Elevated Temperature Stability of Refractory Metal Alloys", First Quarterly Report, WANL-PR-(P)-001.
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- 3. R. T. Begley and L. L. France, "Effect of Oxygen and Nitrogen on Workability and Mechanical Properties of Columbium", ASTM Special Technical Publication 272, 1959.